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UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

ft^3	cubic foot	lb/st	pound per short ton
gal/min	gallon (U.S.) per minute	μm	micrometer
h	hour	mm	millimeter
in	inch	pct	percent
lb	pound	st	short ton
lb/min	pound per minute	wt pct	weight percent

CONTINUOUS FLOTATION TESTING TO RECOVER COBALT FROM MISSOURI LEAD ORES

By W. L. Cornell,[1] A. M. Wethington,[2] D. C. Holtgrefe,[3] and F. H. Sharp[1]

ABSTRACT

The Missouri lead ores are the only domestic sources of cobalt presently being mined and processed for other materials. They represent the best short-term solution for meeting up to 13 pct of the Nation's strategic and critical cobalt needs from domestic sources. Presently, the cobalt is lost as an impurity in the copper, lead, and zinc concentrates and in the mill tailings because the technology to recover it with minium impact on present production does not exist. This paper presents results of research, built upon earlier Bureau of Mines laboratory research, to enumerate the problems encountered in cobalt recovery from the copper concentrate under actual mill conditions; to present alternative solutions to these problems; and finally, to describe the best available technology for cobalt recovery within the imposed constraints.

Mineralogical analyses showed the cobalt present as a distinct mineral phase, predominately siegenite, amenable to a physical separation method such as froth flotation. Flotation was chosen for recovery because of easy adaptation by the mills. Unit processes of grinding, conditioning, and flotation were studied individually and in concert, and a series of continuous tests were run based on the parameters developed to obtain engineering data for subsequent economic analysis. Overall cobalt grades of 3.00 wt pct at a recovery of 64.0 pct were achieved.

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[3]Physical science technician.
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INTRODUCTION

The United States has domestic cobalt resources in Alaska, California, Minnesota, Idaho, and Missouri. The subeconomic nature of the cobalt in these deposits has generally precluded private sector efforts at recovery except in the case of the Blackbird deposit in Idaho and the Madison deposit in Missouri. Because cobalt is a strategic and critical metal, the Bureau of Mines has conducted research aimed at the recovery of cobalt from the Duluth Gabbro Complex[4] of northeastern Minnesota, the laterite deposits along the California-Oregon border,[4] the Madison deposit in Missouri,[4] and the Missouri lead ores of the Viburnum trend.[4]

Of these domestic resources of cobalt, the Missouri lead ores are unique in that they are the only ones being mined commercially at the present time. Economic values of lead, zinc, copper, and silver are recovered at various stages of processing.

The Missouri lead ores account for about 85 pct of the annual primary lead production of the United States, 20 pct of the zinc, 0.8 pct of the copper, and 7 pct of the silver.[5]

Total reserves are in excess of 300 million st and contain about 100 million lb of cobalt and 150 million lb of nickel. On an annual basis, the cobalt mined (but presently not recovered) is equivalent to roughly 13 pct of the U.S. cobalt consumption.[5]

Studies were conducted on ores from the Viburnum trend in southeast Missouri. The Viburnum trend deposits occur along the flank of the St. Francois Mountains. Ores of the Viburnum trend are characteristically complex mixtures of the sulfide minerals galena, PbS; sphalerite, ZnS; chalcopyrite, $CuFeS_2$; pyrite, FeS_2; marcasite, FeS_2; and the cobalt-nickel mineral siegenite, $(Ni,Co)_3S_4$, hosted in a gangue of dolomite, $(Ca,Mg)(CO_3)_2$, with minor inclusions of quartz, SiO_3, and calcite, $CaCO_3$. Minor amounts of other sulfides are also found; these include: bravoite, $(Fe,Ni)S_2$; millerite, NiS; bornite, Cu_5FeS_4; chalcocite, Cu_2S; digenite, $Cu_{2-x}S$; djurleite, $Cu_{1.96}S$; polydymite, Ni_3S_4; vaesite, NiS_2; gersdorffite, NiAsS; tennantite, $(Cu, Fe)_{12}As_4S_{13}$; arsenopyrite, FeAsS; pyrrhotite, $Fe_{1-x}S$; and covellite, CuS.

The major cobalt source in the ore is the mineral siegenite. Separation and concentration of siegenite by froth flotation is complicated by the mineralogy of the ore. The siegenite is often present as an intimate replacement intergrowth with chalcopyrite (fig. 1), sphalerite, galena (fig. 2), and the iron sulfides, pyrite and marcasite. Current grinding practice in the mills tends to leave the siegenite as binary locked particles in the various product streams (fig. 1). Therefore, further grinding to liberate the siegenite particles is required, but is limited by the lower size limitations of the froth flotation and by grinding energy considerations.

During beneficiation, the cobalt is distributed to the various mill product streams as shown in figure 3. The run-of-mine ore contains 0.015 wt pct Co. About 30 pct of this cobalt reports to the copper concentrate at an average grade of 0.50 wt pct Co. This represents an improvement ratio of 33. Because of the significant built-in improvement ratio,

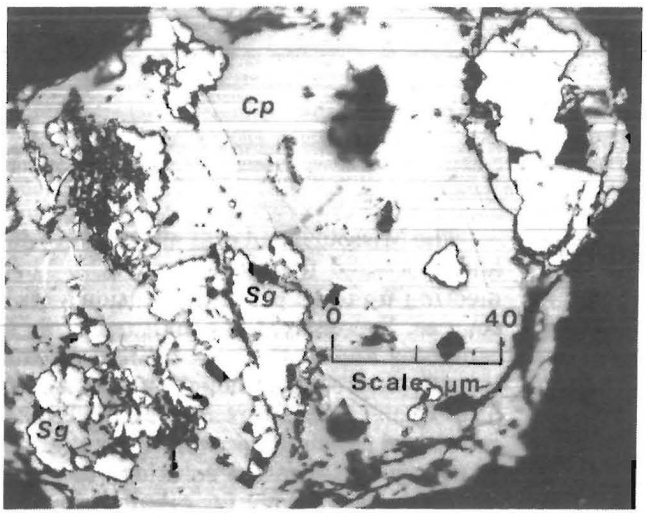

Figure 1.—Chalcopyrite, Cp, particle with siegenite, Sg, inclusions.

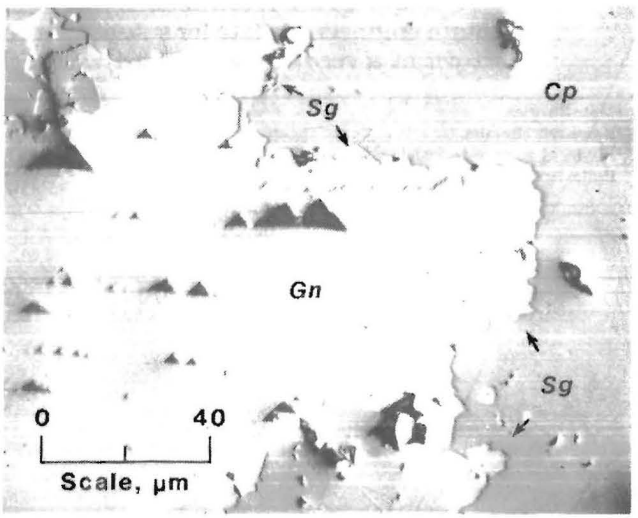

Figure 2.—Galena, Gn, partially coated with siegenite, Sg, imbedded in chalcopyrite, Cp.

[4]Peterson, G. R., D. I. Bleiwas, and P. R. Thomas. Cobalt Availability—Domestic. A Minerals Availability System Appraisal. BuMines IC 8848, 1981, 31 pp.

[5]Kirk, W. S. Cobalt. BuMines Mineral Commodity Profile, 1983, 16 pp.

the copper concentrate was chosen for initial studies, and is the subject of this report.

Previous Bureau of Mines research to recover cobalt from the copper concentrates has included both hydrometallurgical and flotation studies. Hydrometallurgical schemes have utilized a ferric chloride solvent in an oxidative leach to treat the copper concentrate and leave the cobalt in a cobalt-enriched residue. Although successful, this scheme has the disadvantage of disrupting the production of a salable copper product and would involve a major capital investment in a leach and electrowinning plant to recover the copper and the lead from the leach liquor.

In laboratory flotation research, the first attempt was to float the cobalt-bearing siegenite from the chalcopyrite.[6] This proved impossible except for some Halimond tube tests on simple two-mineral systems. Research, therefore, was directed at floating the chalcopyrite from the siegenite.

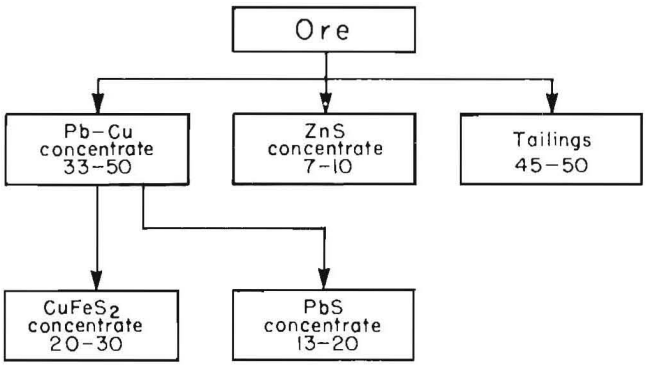

Figure 3.—Distribution, in percent, of cobalt during milling of Missouri lead ores.

ACKNOWLEDGMENTS

The authors would like to thank the management and staff of Cominco American, Inc., and Dresser Industries, Inc., Magmont operation, and especially the mill and maintenance personnel for their assistance in the set up and operation of research equipment at the mill.

MATERIAL, EQUIPMENT, AND PROCEDURES

The original experimental flotation test equipment used at the Cominco American Magmont concentrator consisted of grinding and flotation sections. The grinding section consisted of a 30- by 30-in ball mill in closed circuit with a 3-in hydrocyclone. Feeding was accomplished by a periodic bleed from a continuously circulating stream, from an agitated holding tank. An air-actuated, timed valve accomplished the actual bleed. This feed system gave satisfactory control of parameters. Since the stream was continuously circulating, the feed density was relatively uniform and feed rate could be varied by adjusting the timed cycle on the valve. Two feed points were used. Initially, feed was introduced directly to the ball mill, but the feed point was later changed to the cyclone feed sump. The hydrocyclone was fed from a divided sump by a Vacseal[7] pump. The pump served as a receptacle for both the ball mill discharge and the hydrocyclone overflow and, as mentioned, the feed. The Vacseal pump was charged from a bottom outlet from this cone-shaped sump and the rougher flotation circuit was fed from an overflow near the top of the sump. Provisions were made to feed directly into the No. 1 cell of the flotation section or into an attritioner and then into the No. 1 cell.

The flotation section included six Hazen-Quinn, subaeration cells in the series. These were No. 5 units with an effective volume of 0.25 ft^3 per cell. Later, seven Fagegren type cells were used as roughers. These had an effective volume of 0.20 ft^3 each.

By using three sets of launders on the Hazen-Quinn cells and by altering the flow patterns between the cells, it was possible to vary the flotation configurations.

Figure 4 shows the three of the variations used. In practice, the first pattern was used for roughing and the second for cleaning. The third was tried but was most difficult to control. When in the rougher configuration (pattern 1), concentrates were held in a tank awaiting a change to the second configuration for cleaning. Final tailings from each test were filtered in a pan filter and saved for subsequent testing. All pumping within the unit, except for the cyclone feed, was done by air-driven Warren Rupp Sandpiper pumps equipped with tranquilizer units to smooth the flow.

[6]Clifford, R. K., and L. W. Higley, Jr. Cobalt and Nickel Recovery From Missouri Lead Belt Chalcopyrite Concentrates. BuMines RI 8321, 1978, 14 pp.

[7]Reference to specific products does not imply endorsement by the Bureau of Mines.

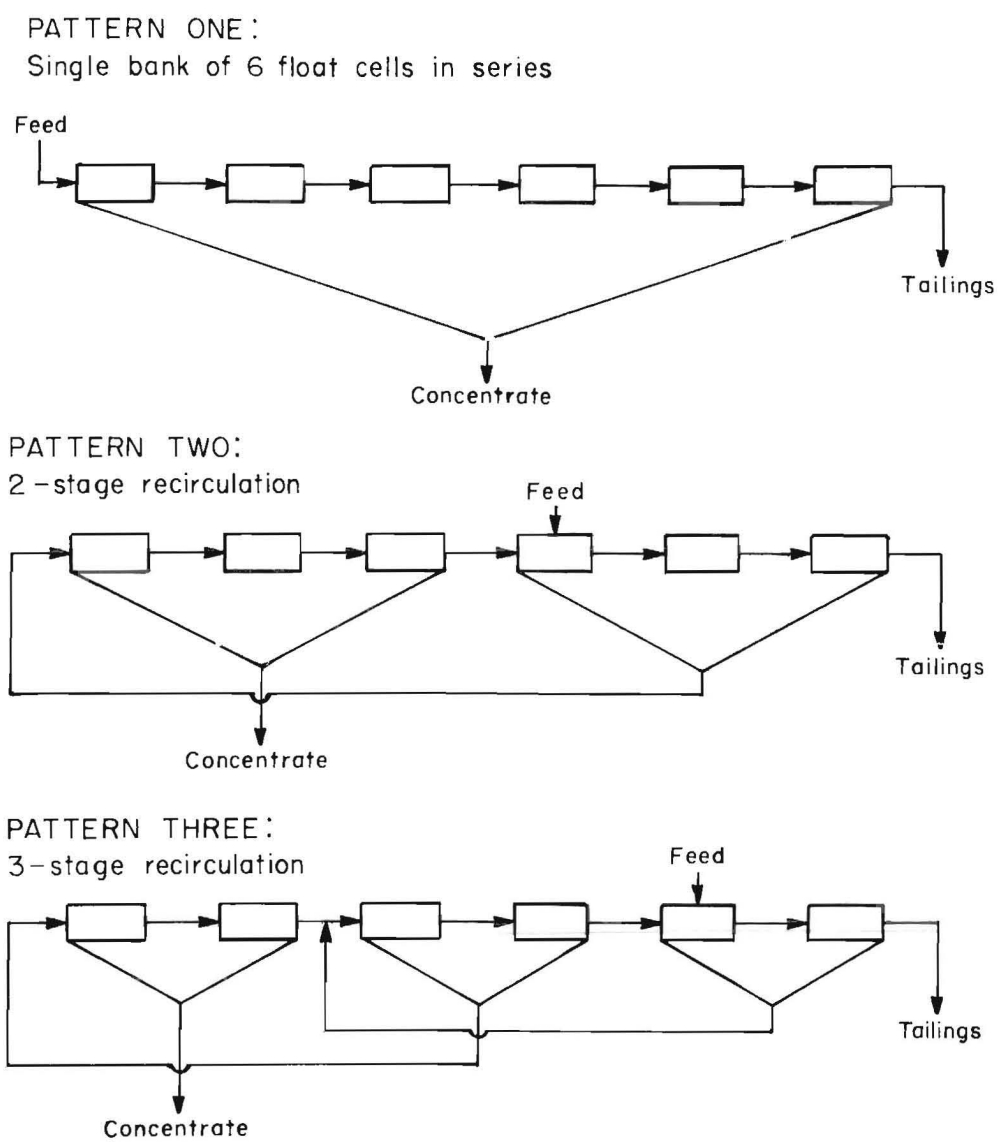

Figure 4.—Cell to cell flow patterns available with experimental flotation test unit.

DESCRIPTION OF FEED MATERIAL

Chalcopyrite concentrate was used as feed stock. A representative long-term chemical analysis of the material is shown in table 1.

Size analyses with assays for each size fraction and distribution of metal values are shown in table 2. It is important to realize there is a high degree of variability in the ore fed into the mill. This can vary day to day or even hour to hour.

Table 1.—Representative chemical analysis of chalcopyrite concentrate

Element	*Assay, wt pct*
Cu	31.00
Fe	29.50
Pb	5.00
Ni	.90
Co	.64
Zn	.60

Table 2.—Cyclosizer analyses and distribution of metal values in a typical $CuFeS_2$ concentrate, weight percent

Screen size, μm	Composition	Assay					
		Co	Cu	Fe	Ni	Pb	Zn
Plus 32	43.9	0.56	29.1	27.0	0.88	8.60	0.63
Minus 32 plus 24	14.6	.51	32.4	30.6	.74	3.90	.49
Minus 24 plus 17	14.8	.44	33.6	30.4	.62	3.70	.38
Minus 17 plus 12	10.9	.36	33.0	30.0	.47	2.70	.26
Minus 12 plus 9	4.5	.26	32.8	29.2	.32	2.50	.20
Minus 9	11.3	.30	34.1	36.3	.43	8.84	.16
Feed	100.0	.47	31.4	29.5	.70	6.30	.46
		Distribution					
		Co	Cu	Fe	Ni	Pb	Zn
Plus 32		52.4	40.7	40.2	55.3	60.0	52.4
Minus 32 plus 24		15.9	15.1	15.2	15.5	9.1	15.9
Minus 24 plus 17		13.8	15.8	15.1	13.0	8.7	13.8
Minus 17 plus 12		8.3	11.4	11.1	7.3	4.7	8.3
Minus 12 plus 9		2.5	4.7	4.5	2.0	1.8	2.5
Minus 9		7.1	12.3	13.9	6.9	15.7	7.1
Feed		100.0	100.0	100.0	100.0	100.0	100.0

DISCUSSION

The continuous flotation testing involved four phases: (1) initial short-duration tests to fix reagent dosages and determine grinding parameters, (2) extended roughing tests, (3) extended roughing tests with successive cleaning stages, and (4) continuous demonstration tests to obtain engineering data. The reagent suite and dosages that were used in prior, large-scale tests were used as a starting point for the continuous flotation tests. These reagents[8] and their respective dosages were sodium aerofloat, 0.50 lb/st; Dowfroth 250 frother, 0.10 lb/st; and sodium cyanide, 0.10 lb/st. All but the frother were added to the ball mill; the frother was added down the cell bank as needed. The grind was targeted at a minimum of 50 wt pct passing 10μm. Prior tests indicated that the siegenite should be sufficiently liberated at this point to give satisfactory rougher recovery.

SHORT-DURATION TESTS

During the short-duration tests, it was discovered that some of the chalcopyrite was of such a refractory nature that additional collection would be needed. Both Minerac "B" and Z-200 proved effective in floating this more refractory fraction of chalcopyrite. The Z-200 was chosen because it was available on site.

The flotation flowsheet developed from these short-duration tests is compared in figure 5 to the classic rougher-scavenger-cleaner configuration. For lack of a better term, this flowsheet was dubbed "successive cleaning." Table 3 shows the results of laboratory cleaning of rougher concentrates produced from a short-duration test. Two successive flotation tests were done in the laboratory at bench scale on a rougher concentrate, and the data from each tailing assay were combined into a hypothetical cobalt product. In each test, recoveries approached or exceeded 80 pct, and the grades exceeded 3.5 wt pct Co on a starting grade of 0.5 wt pct Co. This flotation flowsheet appeared to allow feed grades lower than the previously determined benchmark of 0.8 wt pct Co.[9]

[8]A tabulation of reagents by both manufacturer's designation and by generic designation is given in the appendix.

Table 3.—Results from laboratory cleaning of rougher concentrates from a short-duration test, weight percent cobalt

	Test 1		Test 2	
	Grade	Recovery	Grade	Recovery
Rougher	5.40	34.00	4.60	53.76
First clean	3.00	36.16	1.80	20.73
Second clean	1.90	12.30	1.20	5.10
Combined results	3.83	82.46	3.65	79.59

[9]Sharp, F. H. Lead-Zinc-Copper Separation and Current Practice at the Magmont Mill. Ch. in Flotation, A. M. Gaudin Memorial Volume, ed. by M. C. Fuerstenau. AIME, 1976, pp. 1215-1231.

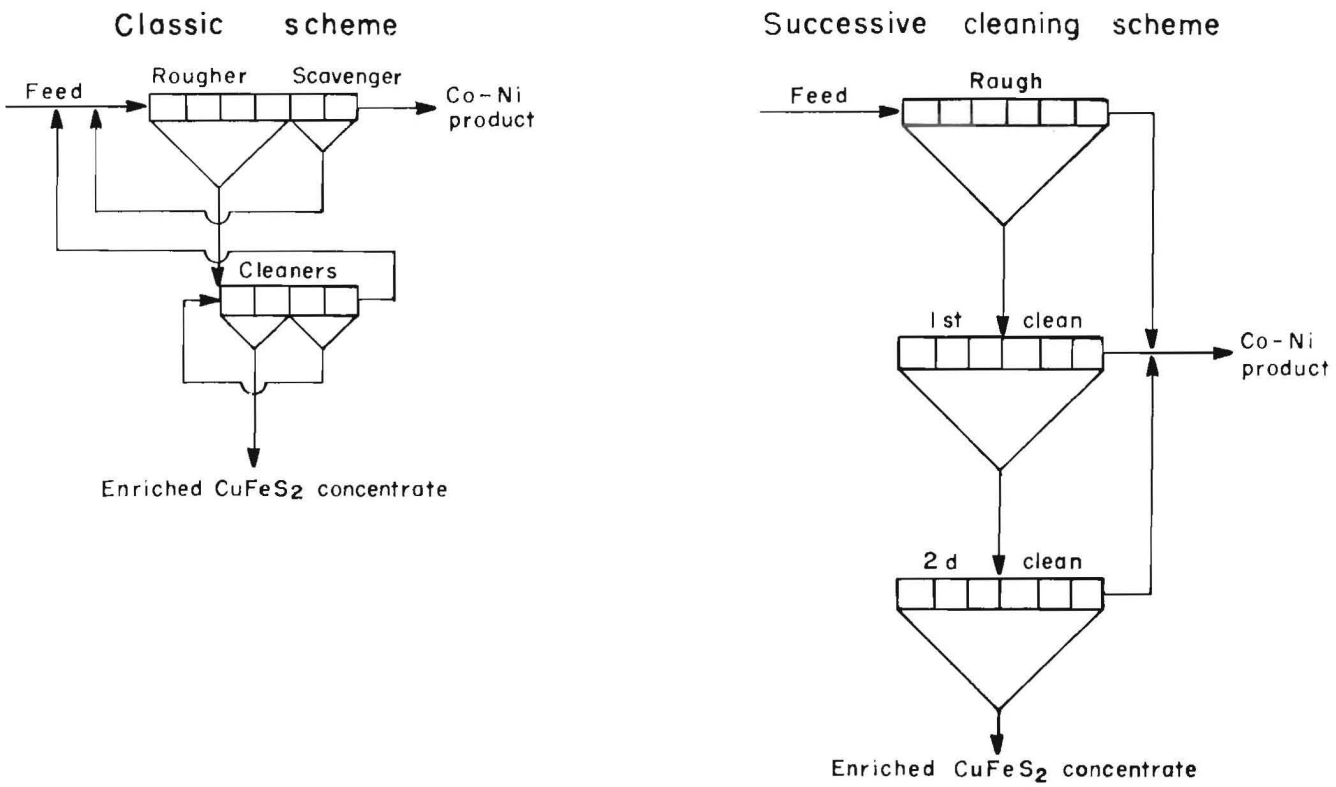

Figure 5.—Schematics of two flotation schemes tested.

EXTENDED ROUGHER TESTS

Extended roughing tests were run to optimize the initial flotation step in order to maximize cobalt grade. These were continuous tests, run around the clock, usually of 3 days to 1 week in duration. Tables 4 and 5 list some typical conditions and data from one of these series of extended tests. Results were evaluated in 8-h periods numbered from one to nine. The objective was to remove 30 to 50 wt pct of the cobalt at as high a grade as possible and leave the successive steps to make a high recovery. As can be seen from table 5, tests 2, 4, 5, and 9 met these standards.

Table 4.—Parameters for extended roughing tests

Test	Reagents, lb/ton			Feed rate, lb/min	Non-$CuFeS_2$ Fe, wt pct
	NaAero	Froth	Z-200		
1	0.51	0.11	0.57	1.54	1.70
2	.54	.11	.60	1.47	1.20
3	.52	.12	.61	1.43	.41
4	.45	.22	.47	1.17	NAp
5	.44	.29	.47	1.17	NAp
6	.54	.72	.56	.99	NAp
7	.54	.66	.52	1.06	3.12
8	.40	.20	.41	1.34	.85
9	.49	.40	.69	1.08	.19

NAp Not applicable.

Table 5.—Results from extended roughing tests, weight percent

Test	Feed assay				Co product assay				Recovery in Co product			
	Co	Cu	Fe	Pb	Co	Cu	Fe	Pb	Co	Cu	Fe	Pb
1	0.77	30.2	28.4	8.5	2.4	14.0	17.9	20.1	37.1	5.6	7.7	26.8
2	.89	30.2	27.9	8.5	5.6	11.0	16.1	15.6	53.8	3.3	5.1	14.9
3	.84	32.0	28.7	7.5	7.0	9.6	17.1	10.4	29.8	1.0	2.1	4.8
4	.82	29.9	26.0	6.3	7.9	8.2	18.5	6.6	31.6	.9	1.1	3.1
5	.88	30.0	26.4	6.7	6.7	9.3	19.1	6.4	43.2	3.6	1.6	5.0
6	.84	32.0	26.9	6.7	8.0	5.2	16.3	4.0	6.6	.1	.4	.4
7	.88	28.6	28.4	6.6	7.5	4.3	12.3	13.9	7.7	.2	.4	1.9
8	.72	31.5	28.7	4.6	8.2	8.4	14.2	4.0	28.2	.6	1.2	2.1
9	.78	31.0	27.6	5.5	4.2	19.8	22.9	7.9	46.1	5.4	7.0	13.9

One very interesting result from these tests was a correlation between the nonchalcopyrite iron (marcasite and pyrite) fraction of the feed and the lead grade of the cobalt product. Earlier work had indicated that there was a relationship between the amount of marcasite-pyrite present in the ore and the extent of galena oxidation.[10] It was also noted that the marcasite was the particularly unstable component. This marcasite-pyrite content seems to fix the galena depression even stronger and makes any significant separation of galena from siegenite very difficult. This leads to an excess of lead in the cobalt product, which both dilutes the cobalt grade and makes further processing to recover the cobalt difficult. As can be seen from table 5, there is no direct correlation between the lead grade of the feed and the lead grade of the cobalt product.

Figure 6 plots lead grade in the cobalt product versus nonchalcopyrite iron in the feed for all continuous tests made with the test unit except for those few made on highly contaminated feedstock (greater than 10 pct Pb in the feed). From these data, two trend lines can be drawn, one with a steeper slope than the other. It is thought that the steeper trend represents a higher marcasite-to-pyrite ratio within the nonchalcopyrite iron fraction.

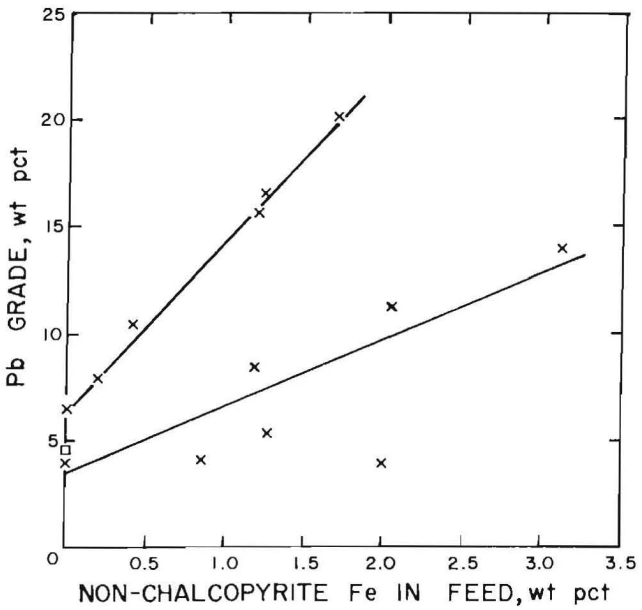

Figure 6.—Lead grade in cobalt product versus nonchalcopyrite iron in the feed for all rougher tests.

[10]Work cited in footnote 9.

EXTENDED TESTS WITH CLEANING

To run the extended roughing tests with cleaning, some modifications to the continuous test unit were necessary.

A large agitation tank was used to hold rougher concentrate, until it was later refloated using a launder configuration that divided the six cells into two sets of three. The tailings from these two steps were added together with the original rougher tailings to form a cobalt product. Tables 6 and 7 list the parameters and summarize the results of a typical test.

Table 6.—Parameters for extended roughing tests with cleaning

	Rough 1	Rough 2
Reagents, lb/st:		
NaAero	0.88	NAp
Froth	0.13	NAp
Z-200	0.38	1.1
Feed rate, lb/min	1.30	1.30
Non-$CuFeS_2$ Fe, wt pct	1.24	2.04

Table 7.—Results from extended roughing and cleaning tests, weight percent

	Rough 1	Rough 2	Clean 1	Clean 2
Feed assay:				
Co	0.43	0.39	0.35	0.36
Cu	29.7	31.4	31.4	32.3
Fe	27.5	29.8	29.3	30.7
Pb	5.0	5.5	4.2	5.1
Co product assay:				
Co	4.8	3.2	.94	1.3
Cu	6.1	16.6	19.0	20.6
Fe	12.3	23.1	16.0	19.8
Pb	16.4	11.2	21.6	20.8
Recovery in Co product:				
Co	15.3	14.1	24.8	40.8
Cu	.3	1.0	5.8	7.4
Fe	.6	1.4	5.5	7.6
Pb	9.5	3.6	32.3	46.1

LIBERATION STUDIES

Figure 7 plots cobalt improvement ratios versus mean particle size for the successive cleaning tests. The improvement ratios show an upward trend with diminishing particle size in the rougher stage but the line is virtually flat for the cleaning stages. This could indicate either a lack of liberation of the siegenite or entrainment of the siegenite in the froth.

Scanning electron microscope (SEM) studies tended to confirm the idea that the liberation of the siegenite from the chalcopyrite was incomplete. These data are summarized in table 8.

The first series of these studies was done in an attempt to determine the degree of liberation of the siegenite from the chalcopyrite. The samples of flotation test unit products

Table 8.—SEM particle distribution of test unit products by chemical characterization and weight percent of sample

Chemical category of mineral	Minus 2 μm			Minus 10 plus 2 μm			Plus 10 μm			Total		
	Cyclone O/F	Cu conc	Co-Ni product	Cyclone O/F	Cu conc	Co-Ni product	Cyclone O/F	Cu conc	Co-Ni product	Cyclone O/F	Cu conc	Co-Ni product
No low X-rays	0.13	0	0.07	0.25	0	0.04	0	0	0	0.38	0	0.06
Co-Ni-Pb	0	0	0	0	0	0	0	0	0	0	0	0
Co-Ni-Mg-Ca	0	0	0	0	0	0	0	0	0	0	0	0
Co-Ni-Cu-Fe	.21	.19	.13	.56	.23	.05	0	0	0	.77	.42	.17
Fe-Co-Ni	.33	.26	1.28	.63	.90	3.60	0	0	0	.96	1.16	4.88
Co-Ni-Zr	0	0	0	0	0	0	0	0	0	0	0	0
Co-Ni	0	0	.03	0	0	.35	0	0	0	0	0	.38
Pb	.66	.99	.46	4.94	1.15	0	0	0	0	5.60	2.14	.46
Mg-Ca	0	.09	.09	0	0	2.91	0	0	3.21	0	.09	6.21
Cu-Fe	10.35	11.72	5.31	34.75	27.65	4.10	32.65	47.7	66.18	77.75	87.07	75.59
Fe-Ni	.02	.01	.08	.29	0	.15	0	0	0	.31	.01	.23
Fe	.50	.34	1.22	.18	0	1.65	0	0	0	.68	.34	2.87
Zn	.05	.02	0	0	0	0	0	0	0	.05	.02	0
Si	.01	.11	.32	.10	0	1.77	0	0	5.47	.11	.11	7.56
Miscellaneous	.40	.67	.83	1.42	.69	.75	11.57	7.28	0	13.39	8.64	1.58
Total	12.66	14.40	9.77	43.12	30.62	15.37	44.22	54.98	74.86	100.00	100.00	100.00

O/F Overflow.

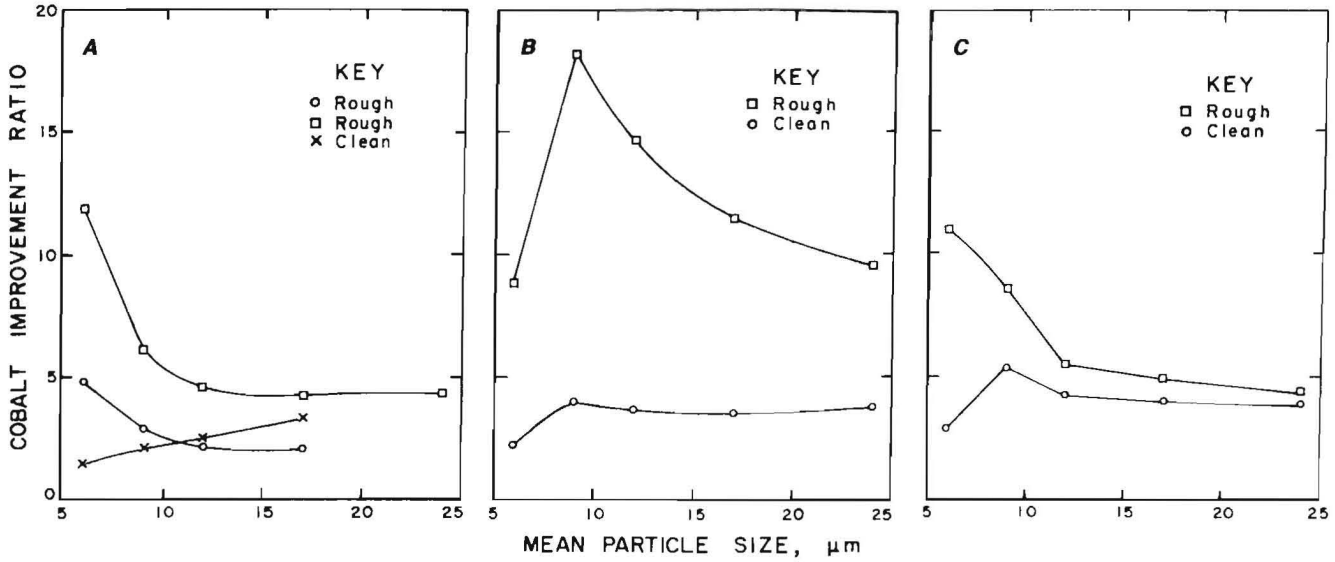

Figure 7.—Cobalt improvement ratio by size fraction for successive cleaning tests. *A*, Early tests; *B*, rough 1 and clean 1; *C*, rough 2 and clean 2.

were searched for nickel by using X-ray mapping. When nickel was found the area was then remapped for cobalt and copper. The occurrence of cobalt and nickel without the copper would indicate a free siegenite particle. Indications were that the siegenite particles were not liberated. Some possible reasons given are that the lack of friability of the particles at this size range makes breakage difficult, or that the lack of distinct phase boundaries and grain structures between the siegenite and chalcopyrite also does not allow liberation of the siegenite.

The second series of studies involved the use of the SEM in conjunction with advanced image analysis and X-ray analysis. Particle distributions of test unit products were done by number of particles, by percent to total particles, and by weight percent of the sample. Again, the conclusion was that although some liberation of siegenite from chalcopyrite occurred, that this liberation involved relatively large composite particles, and that there remained a significant number and weight percent of composite particles in the less than 4-μm fraction.

Although liberation was not complete, the small size of the locked particles ruled out further grinding. Light microscopy studies done of polished sections prepared from the final cleaner concentrate revealed significant numbers of free siegenite particles in the minus 12-μm fraction. This indicated that the entrainment of the siegenite particles in the chalcopyrite froth was also contributing to the cobalt loss (fig. 8).

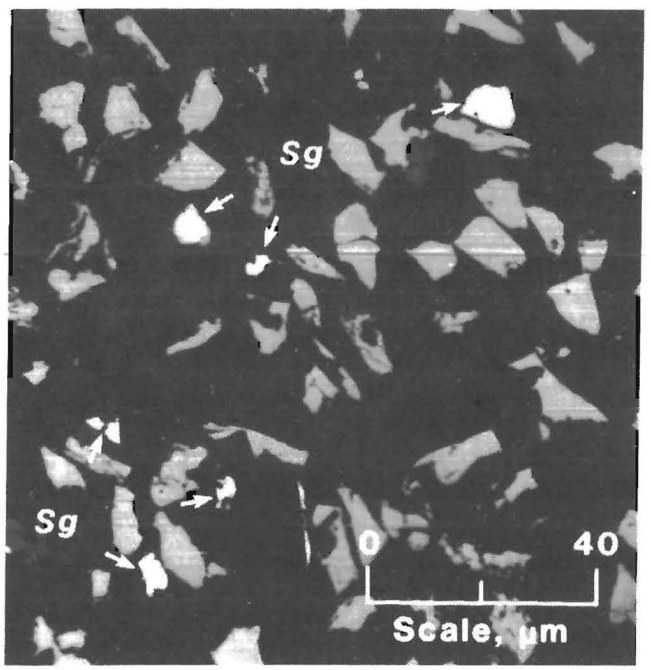

Figure 8.—Cleaner concentrate (minus 12-μm plus 9-μm size fraction) showing free siegenite, Sg, particles entrained in chalcopyrite particles.

ATTRITION GRINDING-CONDITIONING

The copper concentrate is exposed to as many as seven separate reagents prior to cobalt separation. Several methods of reagent removal had been tried previously with little success. These included steaming, Na_2S addition, and oxidation with H_2O_2 and ozone.

It was postulated that attritioning or scrubbing with a ceramic medium might produce a cleaner, more responsive mineral surface with which to work. With this in mind, a series of laboratory tests were initiated to evaluate the effect of attritioning. Bench testing was done with an opposed

impeller scrubber with glass and ceramic media of sizes ranging from 500 to 3,000 μm for 2 h. Table 9 lists sizing data from cyclosizer tests of the attritioned products. For comparison, a typical float cell feed prepared by closed circuit grinding had the distribution in table 10.

Table 9.—Comparison of sizing produced by attritioning with different media, cumulative weight percent passing size

Size, μm	Minus 3- plus 1-mm glass	Minus 1.0- plus 0.7-mm ceramic	Minus 3- plus 1-mm ceramic
32	98.0	79.7	95.1
24	89.5	67.3	86.8
17	64.2	48.4	65.5
12	38.5	31.1	41.9
9	28.1	23.6	31.5

Table 10.—Sizing produced by closed circuit grinding of copper concentrates, cumulative weight percent passing size

Size, μm	
32	93.5
24	92.4
17	86.2
12	65.8
9	51.4

Using the attrition mechanism for primary grinding was ruled out because of energy considerations but it was determined that a scrubber improved flotation results. In particular, the results of tests on attritioned concentrates using no reagents indicated that the mineral surfaces were being favorably altered. Also, these tests gave an indication that the attritioning might remove the starch from the surface of galena and make the galena more responsive. Table 11 shows the results of a test done on attrition-ground copper concentrate with no collector additions and with only a frother used for froth stability. Note in particular the lead assays. There is no upgrading into the tailings, which ran counter to the results on most previous tests.

Table 11.—Results of bench flotation test on attrition-ground copper concentrate using no reagents, weight percent

	Concentrate, 87.7 wt pct	Tails, 12.3 wt pct	Calculated head, 100 wt pct
Assay:			
Co	0.28	1.49	0.43
Cu	26.4	18.2	25.4
Fe	26.2	16.6	25.0
Pb	6.32	4.07	6.04
Distribution:			
Co	57.3	42.6	100.0
Cu	91.2	8.8	100.0
Fe	91.9	8.1	100.0
Pb	91.8	8.2	100.0

CONTINUOUS TESTS FOR ENGINEERING DATA

The final series of tests involved configuring the continuous test unit to obtain engineering data for the flowsheet and parameters developed from the prior tests. In order to be as realistic as possible, the feed for this series of tests was filtered copper concentrate taken from Magmont's storage pad. It was felt that this offered the worst case for feed material and would best simulate the feed of any future circuit to recover cobalt. The concentrate was reslurried in an agitated tank at approximately 60-pct solids. A peristaltic pump was used to feed the slurry to the ball-mill sump at approximately 0.16 gal/min (1.4 lb/min). From the sump, the combined feed and ball mill overflow was pumped to the 3-in cyclone with the underflow going to the mill and the overflow back to the divided ball-mill sump. The overflow then fed by gravity to the attrition machine and from there to the float cells. This setup resulted in a heavy recycling of undersize through the cyclone, giving, in effect, a series of cyclones and resulting in a cyclone efficiency exceeding 97 pct.

By using an agitation holding tank for the concentrates, six stages of flotation were possible. Referring to figure 5 and the successive cleaning scheme, this flowsheet was simply run twice. Typical results from a test with six stages of flotation are given in table 12. Flotation was not carried out beyond six stages, but the grade in the final stage indicates that further stages could be used to increase recovery at little loss of grade. Cobalt grades stage by stage were, by percent: 1, 1.70; 2, 2.10; 3, 2.60; 4, 3.60; 5, 3.40; and 6, 3.00.

For the six-stage tests, the reagents and their dosages were: Sodium Aerofloat, 1.2 lb/st; Z-200, 0.25 lb/st; MIBC, 0.2 lb/st; and mercaptobenzothiozole, 0.5 lb/st. The Sodium Aerofloat was added to the attritioner, the Z-200 to the first stage, the MIBC to the first stage, and the mercaptobenzothiozole to stages 1, 2, 4, and 5.

Table 12.—Overall results of a demonsration test with six stages of sucessive cleaning, weight percent

	Feed	Final conc, 85.2 wt pct	Combined cobalt tails, 14.8 wt pct
Assay:			
Co	0.70	0.30	3.00
Cu	32.60	33.40	21.70
Fe	32.30	32.40	22.50
Ni	.90	.40	4.20
Pb	5.00	4.60	8.50
Zn	1.00	1.00	.86
Distribution:			
Co	100.0	36.0	64.0
Cu	100.0	89.9	10.1
Fe	100.0	89.8	10.2
Ni	100.0	35.4	64.6
Pb	100.0	75.7	24.3
Zn	100.0	87.0	13.0

CONCLUSIONS

Research using a continuous flotation test unit has resulted in a flowsheet that allows the recovery of 70 pct or more of the cobalt in Missouri lead ore copper concentrates, at grades of greater than 3 pct Co or 7 pct combined Ni-Co. This can be accomplished on feeds grading as low as 0.25 pct Co.

The flowsheet developed involves stagewise separations of the cobalt and copper-bearing materials and is more flexible than the classic rough, scavenge, and clean configuration.

APPENDIX

The following tabulation lists reagents by both the manufacturer's designation and by generic designation.

Manufacturer's designation	Generic designation
Dow Z-200	Isopropyl ethyl thionocarbamate.
Cyanimid Sodium Aerofloat	Sodium dialkyl dithiophosphate.
Cyanamid R-404	Sodium mercaptobenzothiazole.
MIBC	Methylisobutyl carbinol.
Oreprep F-507	High-molecular-weight polyol.
Dow DF-250	Polypropylene glycol methyl ether.
Minerac "B"	A dixanthogen formula.

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